UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5 CHICAGO REGIONAL LABORATORY
536 SOUTH CLARK STREET (ML-10C)
CHICAGO, ILLINOIS 60605

ELECTRONIC LABORATORY DATA PACKAGE
FINAL LEVEL IV
COVER PAGE

LIMS Work order(s):	1806004
Analysis:	Ignitability by Setaflash & Water Content, Karl Fisher Titration
Primary Analyst:	Colin Breslin
Date:	07/19/2018
Data Reporting Qualtrax Workflow ID:	15105

Digital Signature of Primary Analyst:

Colin Breslin
Digitally signed by COLIN BRESLIN
Date: 2018.07.19 15:58:51 -05'00'

Digital Signature Agreement: By signing above the primary analyst understands and agrees that they will be held legally bound, obligated, and responsible for the use of their digital signature as they would be by using their hand-written signature.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

Date: **7/19/2018**

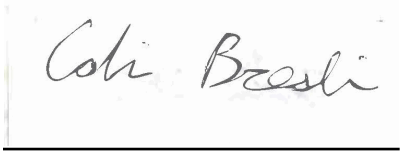
Subject: **Review of Region 5 Data for Milliken Chemical Dewey Plant**

To: **Region 4**
980 College Station Road
Athens, GA 30605

From: **Colin Breslin, Chemist**
US EPA Region 5 Chicago Regional Laboratory

The data transmitted under this cover memo successfully passed CRL's data review procedures as documented in the current Quality Management Plan and applicable Standard Operating Procedures. In accordance with the EPA QA/G-8 *Guidance on Environmental Data Verification and Data Validation* and the U.S. EPA Region 5 RMD QMP, CRL performs data verification on all the data generated internally. CRL does not perform data validation or quality assessment procedures.

This report was reviewed and the information provided herein accurately represents the analysis performed.



Please contact the analyst with any technical report issues, Robert Thompson at (312)-353-9078 for sample project concerns, and Sylvia Griffin at (312)-353-9073 with data transmittal questions. Thank you.

Attached are Results for: **Milliken Chemical Dewey Plant**

Analyses included in this report:

Ignitability by Setaflash

Water content,Karl Fisher Titration

Report Name: 1806004 Ignitability by Setaflash Water content,Karl Fisher Titration FINAL Jul 19 18 1448

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Region 4	Project: Milliken Chemical Dewey Plant	
980 College Station Road	Project Number: [none]	**Reported:**
Athens GA, 30605	Project Manager: Jeffrey Hendel	Jul-19-18 14:48

Accredited Analyses included in this Report

Method: ***EPA 1020B in Water***
Analysis: ***Ignitability by Setaflash***

Analyte	Certifications
Ignitability by Flashpoint	ISO/IEC 17025:2005

Method: ***EPA 9000 in Water***
Analysis: ***Water content,Karl Fisher Titration***

Analyte	Certifications
Water content	ISO/IEC 17025:2005

Analytes not listed above are not accredited by ANAB.

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Region 4	Project: Milliken Chemical Dewey Plant	
980 College Station Road	Project Number: [none]	**Reported:**
Athens GA, 30605	Project Manager: Jeffrey Hendel	Jul-19-18 14:48

Analysis Case Narrative

General Information

Two samples for the analysis of Ignitability by Setaflash (flashpoint) and Water Content, Karl Fisher Titration were received at the Chicago Regional Laboratory (CRL) on June 06, 2018. Holding times do not apply to these samples. The designated analyst, Colin Breslin, can be reached at 312-886-2912.

The flashpoint samples were prepared and analyzed according to CRL SOP AIG048A Version 5 (based on SW-846: 1020B).

The water content samples were prepared and analyzed according to CRL SOP AIG015A Version 4 (based on SW-846: 9000).

Sample Analysis and Results

The data reported herein meets the requirements of the CRL analytical SOP used for analysis. There were no laboratory specifications stated in the Milliken Chemical Dewey Plant Quality Assurance Project Plan (QAPP), dated September 19, 2017. The QAPP is saved on the CRL share drive with file name: Miliken_QAPP_17-0496.pdf.

Water content results were reported as not detected at the CRL analytical SOP reporting limit of 0.5% water content, which showed the samples were essentially non-aqueous.

Quality Control

All Quality Control (QC) audits were within CRL limits for the requested analytes or did not result in qualification of the data.

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

WORK ORDER **Printed: 6/7/2018 11:07:31AM**

1806004

US EPA Region 5 Chicago Regional Laboratory

Client:	**Region 4**	**Project Manager:**	**Angela Ockrassa Davis**
Project:	**Milliken Chemical Dewey Plant**	**Project Number:**	**[none]**

Report To:

Jeffrey Hendel	980 College Station Road	Phone: (706) 355-8839
Region 4	Athens, GA 30605	Fax:

Date Due:	Jul-23-18 15:00 (46 day TAT)		
Received By:	Robert Snyder	Date Received:	Jun-06-18 09:15
Logged In By:	Robert Snyder	Date Logged In:	Jun-06-18 13:57

Samples Received at:	22.8 °C
Sample tags/labels	**Yes**
Seals Intact	**Yes**
Received on ice	**No**
Paperwork Included	**Yes**

Work Order Comments:
Copy/Relog from 1805008.

Sample ID: 1806004-01 **Sampled:** Sep-26-17 12:25 **Matrix:** Water

Sample Name: 01 Tetramer **Sample Location/Comments:** E173905-01

Sample Comments:

Analysis	Hold time (days)	Expires	Comments
Ignitability by Setaflash	365	Sep-26-18 12:25	pH = 4
Water content,Karl Fisher Titration	365	Sep-26-18 12:25	pH = 4

Sample ID: 1806004-02 **Sampled:** Sep-26-17 12:35 **Matrix:** Water

Sample Name: 02 Tetramer **Sample Location/Comments:** E173905-02

Sample Comments:

Analysis	Hold time (days)	Expires	Comments
Ignitability by Setaflash	365	Sep-26-18 12:35	pH = 4
Water content,Karl Fisher Titration	365	Sep-26-18 12:35	pH = 4

WORK ORDER MEMO: Samples rec'd at ambient temperature. Last sample set for this project was also, and NCR 14124 was opened. NCR was resolved as not being an issue, as flash point analysis does not have a requirement to be cooled but the sample requirements table just hadn't been updated yet. Update currently pending, so no NCR will be opened for this shipment.

REVIEWED
By Robert Thompson at 11:08 am, Jun 07, 2018

Reviewed By Date

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Region 4	Project: Milliken Chemical Dewey Plant	
980 College Station Road	Project Number: [none]	**Reported:**
Athens GA, 30605	Project Manager: Jeffrey Hendel	Jul-19-18 14:48

Water Content by SW846 Method 9000
US EPA Region 5 Chicago Regional Laboratory

01 Tetramer (1806004-01) **Matrix: Water** **Sampled: Sep-26-17 12:25** **Received: Jun-06-18 09:15**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Water content	**U**			0.5	%	1	B18F011	Jun-12-18	Jun-12-18

02 Tetramer (1806004-02) **Matrix: Water** **Sampled: Sep-26-17 12:35** **Received: Jun-06-18 09:15**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Water content	**U**			0.5	%	1	B18F011	Jun-12-18	Jun-12-18

Environmental Protection Agency Region 5
Chicago Regional Laboratory

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Region 4	Project: Milliken Chemical Dewey Plant	
980 College Station Road	Project Number: [none]	**Reported:**
Athens GA, 30605	Project Manager: Jeffrey Hendel	Jul-19-18 14:48

Ignitability, Flash Point, EPA 1020B (modified)
US EPA Region 5 Chicago Regional Laboratory

01 Tetramer (1806004-01) **Matrix: Water** **Sampled: Sep-26-17 12:25** **Received: Jun-06-18 09:15**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Ignitability by Flashpoint	**136**				Degrees F	1	B18F004	Jun-08-18	Jun-08-18

02 Tetramer (1806004-02) **Matrix: Water** **Sampled: Sep-26-17 12:35** **Received: Jun-06-18 09:15**

Analyte	Result	Flags / Qualifiers	MDL	Reporting Limit	Units	Dilution	Batch	Prepared	Analyzed
Ignitability by Flashpoint	**136**				Degrees F	1	B18F004	Jun-08-18	Jun-08-18

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Region 4	Project: Milliken Chemical Dewey Plant	
980 College Station Road	Project Number: [none]	**Reported:**
Athens GA, 30605	Project Manager: Jeffrey Hendel	Jul-19-18 14:48

Notes and Definitions

U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Quality Control Summary

Date: 7/19/2018 **Analyst:** Colin Breslin

Project: Milliken Chemical Dewey Plant

Analyte: Ignitability by Flashpoint

Analysis: Ignitability by Setaflash

QC Sample Name	Result	Units	Flags / Qualifiers	MDL	Reporting Limit	Spike Level	Source Result	%REC	%REC Limits	% RPD	% RPD Limit
B18F004-DUP2 Source: 1806004-01	**136**	Degrees F					136			0.0147	2.23
B18F004-SRM1	**80.0**	Degrees F				81.00		98.8%	98.1-101.9		
B18F004-SRM2	**81.0**	Degrees F				81.00		100%	98.1-101.9		
B18F004-SRM3	**80.6**	Degrees F	CustomFlag			81.00		99.4%	98.1-101.9		
B18F004-SRM4	**81.0**	Degrees F				81.00		100%	98.1-101.9		
B18F004-SRM5	**81.0**	Degrees F				81.00		100%	98.1-101.9		
B18F004-SRM6	**81.0**	Degrees F	CustomFlag			81.00		100%	98.1-101.9		
B18F004-SRM7	**80.8**	Degrees F				81.00		99.7%	98.1-101.9		
B18F004-SRM8	**79.7**	Degrees F				81.00		98.4%	98.1-101.9		
B18F004-SRM9	**80.2**	Degrees F	CustomFlag			81.00		99.1%	98.1-101.9		

Analyte: Water content

Analysis: Water content,Karl Fisher Titration

QC Sample Name	Result	Units	Flags / Qualifiers	MDL	Reporting Limit	Spike Level	Source Result	%REC	%REC Limits	% RPD	% RPD Limit
B18F011-DUP7 Source: 1806004-01	**0.01**	%			0.5		0.01			0.00	10
B18F011-DUP8 Source: 1806004-02	**0.01**	%			0.5		0.01			0.00	10
B18F011-SRM1	**1.04**	%				1.000		104%	97-107		
B18F011-SRM2	**1.04**	%				1.000		104%	97-107		
B18F011-SRM3	**1.03**	%				1.000		103%	97-107		
B18F011-SRM4	**1.04**	%				1.000		104%	97-107		

Report Name: 1806004 Ignitability by Setaflash Water content,Karl Fisher Titration FINAL Jul 19 18 1436

Environmental Protection Agency Region 5
Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Quality Control Summary

Date: 7/19/2018 **Analyst:** Colin Breslin

Project: Milliken Chemical Dewey Plant

Notes and Definitions

CustomFlag	B18F004-SRM9: average of B18F004-SRM7 & B18F004-SRM8
CustomFlag	B18F004-SRM6: average of B18F004-SRM4 & B18F004-SRM5
CustomFlag	B18F004-SRM3: average of B18F004-SRM1 & B18F004-SRM2
U	Not Detected
NR	Not Reported
Q	QC limit Exceeded

REVIEWED
By Colin Breslin at 2:44 pm, Jul 19, 2018

Colin Breslin, Chemist

Environmental Protection Agency Region 5

Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

WORK ORDER

1806004

Printed: 6/7/2018 11:07:31AM

US EPA Region 5 Chicago Regional Laboratory

Client: Region 4
Project: Milliken Chemical Dewey Plant
Project Manager: Angela Ockrassa Davis
Project Number: [none]

Report To:

Jeffrey Hendel	980 College Station Road	Phone: (706) 355-8839
Region 4	Athens, GA 30605	Fax:

Date Due:	Jul-23-18 15:00 (46 day TAT)		
Received By:	Robert Snyder	Date Received:	Jun-06-18 09:15
Logged In By:	Robert Snyder	Date Logged In:	Jun-06-18 13:57

Samples Received at:	22.8 °C
Sample tags/labels	**Yes**
Seals Intact	**Yes**
Received on ice	**No**
Paperwork Included	**Yes**

Work Order Comments:
Copy/Relog from 1805008.

Sample ID: 1806004-01 **Sampled:** Sep-26-17 12:25 **Matrix:** Water

Sample Name: 01 Tetramer **Sample Location/Comments:** E173905-01

Sample Comments:

Analysis	Hold time (days)	Expires	Comments
Ignitability by Setaflash	365	Sep-26-18 12:25	pH = 4
Water content,Karl Fisher Titration	365	Sep-26-18 12:25	pH = 4

Sample ID: 1806004-02 **Sampled:** Sep-26-17 12:35 **Matrix:** Water

Sample Name: 02 Tetramer **Sample Location/Comments:** E173905-02

Sample Comments:

Analysis	Hold time (days)	Expires	Comments
Ignitability by Setaflash	365	Sep-26-18 12:35	pH = 4
Water content,Karl Fisher Titration	365	Sep-26-18 12:35	pH = 4

WORK ORDER MEMO: Samples rec'd at ambient temperature. Last sample set for this project was also, and NCR 14124 was opened. NCR was resolved as not being an issue, as flash point analysis does not have a requirement to be cooled but the sample requirements table just hadn't been updated yet. Update currently pending, so no NCR will be opened for this shipment.

REVIEWED
By Robert Thompson at 11:08 am, Jun 07, 2018

Reviewed By Date

Breslin, Colin

From: Qualtrax <noreply@qualtraxcloud.com>
Sent: Tuesday, July 17, 2018 8:19 AM
To: Breslin, Colin
Subject: Notification of Client Contact "WO 1806004 Flashpoint" (ID 15093)

The following is a summary of Client Contact Workflow ID 15093. Adjust your project plan accordingly.

Contact Date	Client Name and Affiliation	Project and/or Work Order	Contact Description	Action Request
5/30/2018 12:00:00 AM	US EPA R4 / Jeffrey Hendel	Milliken Chemical Dewey Plant / 1806004	request to send flashpoint samples.	agreed to accept samples.

Breslin, Colin

From: Awanya, Francis
Sent: Wednesday, May 30, 2018 2:05 PM
To: Thompson, Robert A.; Breslin, Colin; Persoon, Carolyn; Schupp, George
Subject: RE: request form R4 flashpoint

Follow Up Flag: Follow up
Flag Status: Flagged

Okay Rob.

Francis A. Awanya
Chemist
Chicago Regional Laboratory
USEPA Region 5
536 South Clark Street (ML-10C)
Chicago, IL 60605
Telephone: (312)886-3682
Fax: (312)692-2404

From: Thompson, Robert A.
Sent: Wednesday, May 30, 2018 1:55 PM
To: Breslin, Colin <breslin.colin@epa.gov>; Awanya, Francis <awanya.francis@epa.gov>; Persoon, Carolyn <persoon.carolyn@epa.gov>; Schupp, George <schupp.george@epa.gov>
Subject: FW: request form R4 flashpoint

2 flashpoint samples from R4 requested. They wanted 30-days. Okay to accept? 250mL poly?

Robert Thompson, MPH
Chemist|Sample Coordinator
U.S.EPA R5 Chicago Regional Laboratory
536 S. Clark St. ML-10C | Chicago, IL 60605
(312) 353-9078 (Direct) | (312) 353-9096 (Fax)

From: Hendel, Jeffrey
Sent: Wednesday, May 30, 2018 1:49 PM
To: Thompson, Robert A. <Thompson.Robert@epa.gov>
Subject: RE: request form

Here is the completed form. Please let me know how much sample that the lab requires for performing flashpoint.

Thanks for your help,
Jeff

Jeffrey R. Hendel

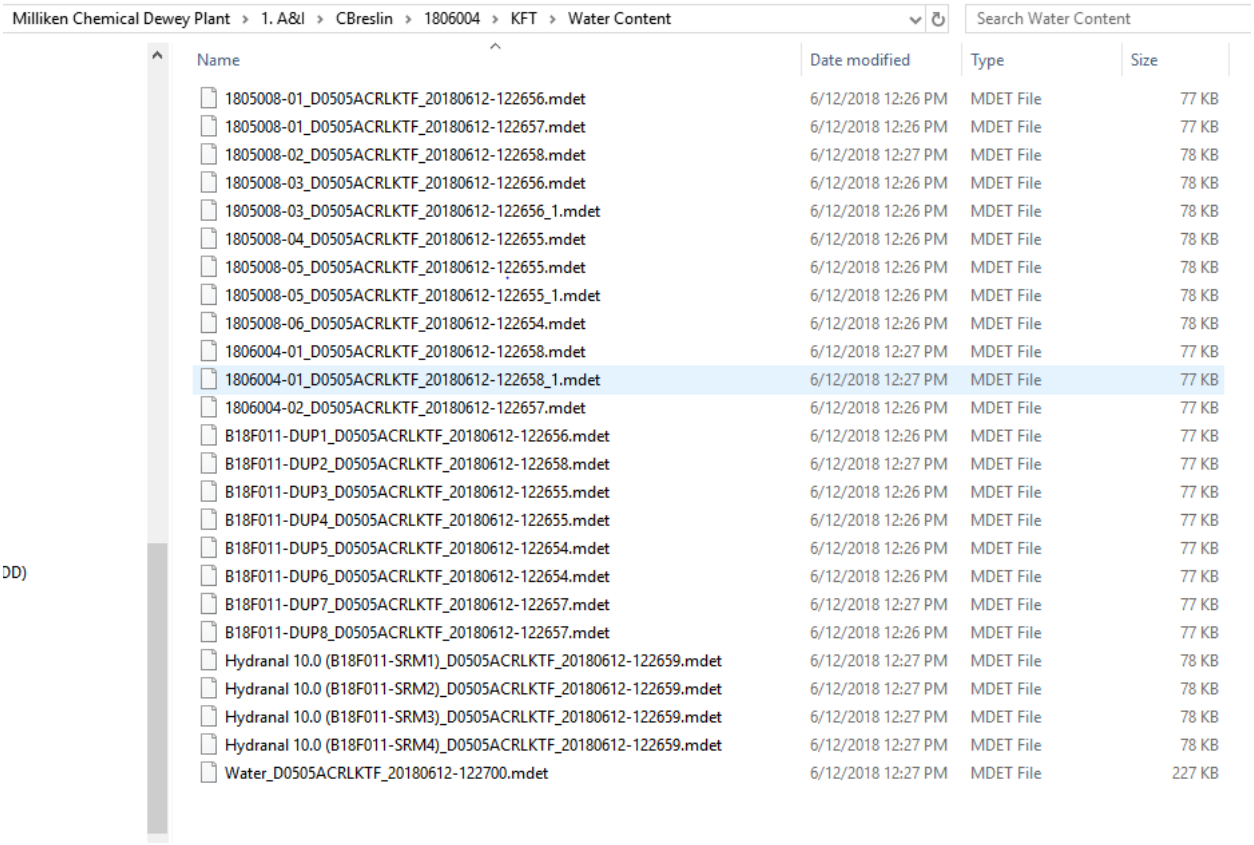
Chief, Inorganic Chemistry Section
U.S. Environmental Protection Agency
980 College Station Road
Athens, GA 30605
Direct: 706-355-8839
Cell: 706-340-2145
hendel.jeffrey@epa.gov

From: Thompson, Robert A.
Sent: Wednesday, May 30, 2018 2:08 PM
To: Hendel, Jeffrey <Hendel.Jeffrey@epa.gov>
Subject: request form

Robert Thompson, MPH
Chemist|Sample Coordinator
U.S.EPA R5 Chicago Regional Laboratory
536 S. Clark St. ML-10C | Chicago, IL 60605
(312) 353-9078 (Direct) | (312) 353-9096 (Fax)

Work Order: 1806004
Analysis: Water Content, Karl Fisher Titration

I:\Projects (New File Structure)\Milliken Chemical Dewey Plant\1. A&I\CBreslin\1806004\KFT\Water Content



Date: 7/17/2018

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

Data Verification Checklist

Work Order: 1806004
Analysis: Ignitability by Setaflash
Project: Milliken Chemical Dewey Plant
CRL SOP Used: AIG048A Version 5

Electronic Pathway(s):
I:\Projects (New File Structure)\Milliken Chemical Dewey Plant\1. A&I\CBreslin\1806004\Setaflash 3\flashpoint

File Name(s):
Not Applicable

General Information

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
1	Did samples meet the laboratory's standard conditions of sample acceptability upon receipt?	YES	YES / NO / NA
2	Was customer contact communication included?	YES	YES / NO / NA
3	Are all field sample ID numbers cross-referenced to the laboratory ID numbers?	YES	YES / NO / NA
4	Were all samples prepared and analyzed within holding times?	NA	YES / NO / NA

Sample Results

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
5	Were calculations checked?	YES	YES / NO / NA
6	Were all analyte results checked?	YES	YES / NO / NA
7	Were all results for soil and sediment samples reported on a dry weight basis?	NA	YES / NO / NA
8	Were % moisture (or solids) reported for all soil and sediment samples?	NA	YES / NO / NA
9	Other than those results < RL, were all other raw values bracketed by calibration standards?	YES	YES / NO / NA
10	Are the RLs for each method analyte included in the laboratory data package?	NA	YES / NO / NA
11	Are MDLs/RLs adjusted for dilutions?	NA	YES / NO / NA
12	Were the raw data (for example, chromatograms, spectral data) reviewed?	YES	YES / NO / NA
13	Were data associated with manual integrations flagged on the raw data?	NA	YES / NO / NA

Standards

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
14	Are all standards used in the analyses NIST-traceable or obtained from other appropriate sources as specified in the analytical SOP?	YES	YES / NO / NA
15	Were standard/reagent preparations checked (preparation date, expiration date, parent standard IDs, etc.), if applicable?	YES	YES / NO / NA

Batch Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
16	Were appropriate type(s) of blanks analyzed?	NA	YES / NO / NA

Batch Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
17	Were blanks analyzed at the appropriate frequency?	NA	YES / NO / NA
18	Were method blanks taken through the entire analytical process, including preparation and, if applicable, cleanup procedures?	NA	YES / NO / NA
19	Were blank concentrations <= MDL or RL, as applicable in the analytical SOP?	NA	YES / NO / NA
20	Was each LCS/SRM taken through the entire analytical procedure, including prep and cleanup steps?	YES	YES / NO / NA
21	Were LCSs/SRMs analyzed at the required frequency?	YES	YES / NO / NA
22	Were LCS/SRM %Rs within the laboratory QC limits or other acceptance criteria?	YES	YES / NO / NA
23	Were the project/method specified analytes included in the MS?	NA	YES / NO / NA
24	Were MS analyzed at the appropriate frequency?	NA	YES / NO / NA
25	Were MS %Rs within the laboratory QC limits?	NA	YES / NO / NA
26	Were appropriate analytical duplicates analyzed for each matrix?	YES	YES / NO / NA
27	Were analytical duplicates analyzed at the appropriate frequency?	YES	YES / NO / NA
28	Were RPDs or relative standard deviations within the laboratory QC limits or other acceptance criteria?	YES	YES / NO / NA
29	Were RLs analyzed at the appropriate frequency?	NA	YES / NO / NA
30	Were RL recoveries within the laboratory QC limits?	NA	YES / NO / NA

Calibration

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
31	Were calibration correlation coefficient or other acceptance criteria met?	YES	YES / NO / NA
32	Was the number of calibration standards recommended in the method used for all analytes?	NA	YES / NO / NA
33	Were all points generated between the lowest and highest standard used to calculate the curve?	NA	YES / NO / NA
34	Are calibration data available for all instruments used?	YES	YES / NO / NA

Calibration Verification

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
35	Was the absolute value of the analyte concentration in the ICB/CCB <= MDL or RL, as applicable in the analytical SOP?	NA	YES / NO / NA
36	Was the calibration curve verified for each analyte?	NA	YES / NO / NA
37	Has the calibration curve been verified using an appropriate second source standard?	NA	YES / NO / NA
38	Were ICV/CCV analyzed at the method-required frequency?	NA	YES / NO / NA
39	Were ICV/CCV %R within the laboratory QC limits?	NA	YES / NO / NA

Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
40	QC limits in LIMS checked against the SOP?	YES	YES / NO / NA
41	Were percent differences, recoveries, and the linearity within the QC limits specified in the method?	YES	YES / NO / NA

Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
42	Were QC charts checked if a QC audit, excluding matrix QC (MS or DUP), was out of limit?	NA	YES / NO / (NA)

Supporting Data

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
43	Were support equipment data (balance verification, data logs, logbook entries, etc.) included in data package?	YES	(YES) / NO / NA
44	Were approved spreadsheet(s) used?	YES	(YES) / NO / NA

Document Verification

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
45	Is the MDL or RL study up-to-date for each reported analyte?	NA	YES / NO / (NA)
46	Is documentation of the analyst's capability up-to-date and on file? ID 14749	YES	(YES) / NO / NA
47	Are the procedures for compound/analyte identification documented?	YES	(YES) / NO / NA
48	Are laboratory SOPs current and on file for the method performed?	YES	(YES) / NO / NA
49	Were data evaluated against project QAPP or Sample Plan and documented in case narrative?	YES	(YES) / NO / NA

Accreditation

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
50	ANAB logo used appropriately?	YES	(YES) / NO / NA

Signature and Date: REVIEWED By Colin Breslin at 9:33 am, Jul 17, 2018 REVIEWED By Francis A Awanya at 1:05 pm, Jul 18, 2018

Date: 7/17/2018

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5 CHICAGO REGIONAL LABORATORY

536 SOUTH CLARK STREET

CHICAGO, ILLINOIS 60605

Data Verification Checklist

Work Order: 1806004
Project: Milliken Chemical Dewey Plant
Analysis: Water content,Karl Fisher Titration
CRL SOP Used: AIG015A Version 4

Electronic Pathway(s):
I:\Projects (New File Structure)\Milliken Chemical Dewey Plant\1. A&I\CBreslin\1806004\KFT\Water Content

File Name(s):
See Memo-to-File: Data Archive Paths

General Information

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
1	Did samples meet the laboratory's standard conditions of sample acceptability upon receipt?	YES	[YES] NO / NA
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3	Are all field sample ID numbers cross-referenced to the laboratory ID numbers?	YES	[YES] NO / NA
4	Were all samples prepared and analyzed within holding times?	NA	YES / NO / [NA]

Sample Results

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
5	Were calculations checked?	YES	[YES] NO / NA
6	Were all analyte results checked?	YES	[YES] NO / NA
7	Were all results for soil and sediment samples reported on a dry weight basis?	NA	YES / NO / [NA]
8	Were % moisture (or solids) reported for all soil and sediment samples?	NA	YES / NO / [NA]
9	Other than those results < RL, were all other raw values bracketed by calibration standards?	YES	[YES] NO / NA
10	Are the RLs for each method analyte included in the laboratory data package?	YES	[YES] NO / NA
11	Are MDLs/RLs adjusted for dilutions?	NA	YES / NO / [NA]
12	Were the raw data (for example, chromatograms, spectral data) reviewed?	YES	[YES] NO / NA
13	Were data associated with manual integrations flagged on the raw data?	NA	YES / NO / [NA]

Standards

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14	Are all standards used in the analyses NIST-traceable or obtained from other appropriate sources as specified in the analytical SOP?	YES	[YES] NO / NA
15	Were standard/reagent preparations checked (preparation date, expiration date, parent standard IDs, etc.), if applicable?	YES	[YES] NO / NA

Batch Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
16	Were appropriate type(s) of blanks analyzed?	NA	YES / NO / [NA]

Batch Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
17	Were blanks analyzed at the appropriate frequency?	NA	YES / NO / NA
18	Were method blanks taken through the entire analytical process, including preparation and, if applicable, cleanup procedures?	NA	YES / NO / NA
19	Were blank concentrations <= MDL or RL, as applicable in the analytical SOP?	NA	YES / NO / NA
20	Was each LCS/SRM taken through the entire analytical procedure, including prep and cleanup steps?	YES	YES / NO / NA
21	Were LCSs/SRMs analyzed at the required frequency?	YES	YES / NO / NA
22	Were LCS/SRM %Rs within the laboratory QC limits or other acceptance criteria?	YES	YES / NO / NA
23	Were the project/method specified analytes included in the MS?	NA	YES / NO / NA
24	Were MS analyzed at the appropriate frequency?	NA	YES / NO / NA
25	Were MS %Rs within the laboratory QC limits?	NA	YES / NO / NA
26	Were appropriate analytical duplicates analyzed for each matrix?	YES	YES / NO / NA
27	Were analytical duplicates analyzed at the appropriate frequency?	YES	YES / NO / NA
28	Were RPDs or relative standard deviations within the laboratory QC limits or other acceptance criteria?	YES	YES / NO / NA
29	Were RLs analyzed at the appropriate frequency?	NA	YES / NO / NA
30	Were RL recoveries within the laboratory QC limits?	NA	YES / NO / NA

Calibration

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
31	Were calibration correlation coefficient or other acceptance criteria met?	YES	YES / NO / NA
32	Was the number of calibration standards recommended in the method used for all analytes?	NA	YES / NO / NA
33	Were all points generated between the lowest and highest standard used to calculate the curve?	NA	YES / NO / NA
34	Are calibration data available for all instruments used?	YES	YES / NO / NA

Calibration Verification

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
35	Was the absolute value of the analyte concentration in the ICB/CCB <= MDL or RL, as applicable in the analytical SOP?	NA	YES / NO / NA
36	Was the calibration curve verified for each analyte?	NA	YES / NO / NA
37	Has the calibration curve been verified using an appropriate second source standard?	NA	YES / NO / NA
38	Were ICV/CCV analyzed at the method-required frequency?	NA	YES / NO / NA
39	Were ICV/CCV %R within the laboratory QC limits?	NA	YES / NO / NA

Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
40	QC limits in LIMS checked against the SOP?	YES	YES / NO / NA
41	Were percent differences, recoveries, and the linearity within the QC limits specified in the method?	YES	YES / NO / NA

Quality Control

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
42	Were QC charts checked if a QC audit, excluding matrix QC (MS or DUP), was out of limit?	NA	YES / NO / (NA)

Supporting Data

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
43	Were support equipment data (balance verification, data logs, logbook entries, etc.) included in data package?	YES	(YES) NO / NA
44	Were approved spreadsheet(s) used?	YES	(YES) NO / NA

Document Verification

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
45	Is the MDL or RL study up-to-date for each reported analyte?	NA	YES / NO / (NA)
46	Is documentation of the analyst's capability up-to-date and on file? ID 15096	YES	(YES) NO / NA
47	Are the procedures for compound/analyte identification documented?	YES	(YES) NO / NA
48	Are laboratory SOPs current and on file for the method performed?	YES	(YES) NO / NA
49	Were data evaluated against project QAPP or Sample Plan and documented in case narrative?	YES	(YES) NO / NA

Accreditation

Question #	Question	Analyst Response (YES/ NO/ NA)	Reviewer Response (Circle Response)
50	ANAB logo used appropriately?	YES	(YES) NO / NA

Signature and Date: REVIEWED By Colin Breslin at 10:38 am, Jul 17, 2018 REVIEWED By Francis A Awanya at 1:06 pm, Jul 18, 2018

Environmental Protection Agency Region 5

Chicago Regional Laboratory

536 South Clark Street, Chicago, IL 60605
Phone:(312)353-8370 Fax:(312)886-2591

Date: 6/13/2018

AUTOMATED ANALYZER BENCH SHEET

Batch Number: B18F004

Analyses Included On This Benchsheet

Ignitability by Setaflash

Date: 6/13/2018

Analyst (initials): CB

Sample Number	Source ID	Spike Type	Spike ID	Spike Amount (uL)	Final Volume (mL)	Dilution (mL)		Comments
						Initial	Final	
1805008-01								pH = 4
1805008-02								pH = 4
1805008-03								pH = 7
1805008-04								pH = 7
1805008-05								pH = 4
1805008-06								pH = 9
1806004-01								pH = 4
1806004-02								pH = 4
B18F004-DUP1	1805008-02							
B18F004-DUP2	1806004-01							
B18F004-SRM1		Static	17E2502		2			
B18F004-SRM2		Static	17E2502		2			
B18F004-SRM3		Static	17E2502		2			avg of SRM1 & SRM2

Batch Comments:

Environmental Protection Agency Region 5

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Date: 6/13/2018

AUTOMATED ANALYZER BENCH SHEET

Batch Number: B18F004

Date: 6/13/2018

Analyst (initials): CB

Analyses Included On This Benchsheet

Ignitability by Setaflash

Sample Number	Source ID	Spike Type	Spike ID	Spike Amount (uL)	Final Volume (mL)	Dilution (mL)		Comments
						Initial	Final	
B18F004-SRM4		Static	17E2502		2			
B18F004-SRM5		Static	17E2502		2			
B18F004-SRM6		Static	17E2502		2			avg of SRM4 & SRM5
B18F004-SRM7		Static	17E2502		2			
B18F004-SRM8		Static	17E2502		2			
B18F004-SRM9		Static	17E2502		2			avg of SRM7 & SRM8

PREPARATION REAGENTS/STANDARDS/PIPETTES:

17E2502:	**PARAXYLENE FLASH POINT CHECK**	**Prepared: May-25-17**	**Expires: May-25-27**
17C1307:	**Barometer - Digital**	**Prepared: Mar-13-17**	**Expires: Feb-03-19**

Batch Comments:

Flash Point by Setaflash **Working Bench Sheet**

Work Order(s): 1805008 Date: 6/6/18 Analyst: CB

LIMS Batch ID: B18F004

Sample ID: SRM, LIMS # 17E2502
B18F004-SRM1

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
78	NO	990 mb
79	Yes	990 mb

Sample ID: SRM, LIMS # 17E2502
B18F004-SRM2

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
76	NO	990 mb
77	NO	990 mb
78	NO	990 mb
79	NO	990 mb
80	Yes	990 mb

Sample ID: 1805008-01

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
77	NO	991 mb
86	NO	991 mb
95	NO	991 mb
104	NO	991 mb
113	NO	991 mb
122	NO	991 mb
131	NO	992 mb
140	NO	992 mb
149	NO	992 mb
140	NO	991 mb
149	NO	991 mb

Sample ID: 1805008-01

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

no Finite. No Flash observed during range Finding. -CB 6/6/18

Flash Point by AIG048A Date: 6/6/18 Analyst: CB

Continuation from previous page CB 6/6/18

Sample ID: ~~1505~~ 1805008-02

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
78	NO	990 mb
87	NO	990 mb
96	NO	990 mb
105	NO	990 mb
114	NO	990 mb
123	NO	991 mb
132	NO	991 mb
141	NO	991 mb
149	yes	991 mb

Sample ID: 1805008-02

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
141	NO	991 mb
142	NO	991 mb
143	NO	991 mb
144	NO	991 mb
145	Yes	991 mb
143	NO	991 mb
144	NO	990 mb
145	NO	990 mb
146	Yes	990 mb

Sample ID: _____

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

Range Finding NA

For duplicate.

-CB 6/6/18

Sample ID: B18F004-DUP1 (Source = 1805008-02)

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
141	NO	989 mb
142	NO	989 mb
143	NO	989 mb
144	NO	988 mb
145	NO	988 mb
146	NO	988 mb
147	Yes	988 mb
144	NO	988 mb
145	NO	989 mb
146	NO	989 mb
147	Yes	988 mb

Flash Point by Setaflash **Working Bench Sheet**

Work Order(s): 1805008 Date: 6/7/18 Analyst: CB

LIMS Batch ID: B18F064

Sample ID: SRM, LIMS # 17E2502
B18F004-SRM4

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
75	NO	990mb
76	NO	990mb
77	NO	990mb
78	NO	990mb
79	NO	990mb
80	Yes	990mb

Sample ID: SRM, LIMS # 17E2502
B18F004-SRM5

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
78	NO	990mb
79	NO	990 mb
80	Yes	990 mb

Sample ID: 1805008-02 03 CB 6/7/18

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
80	NO	990 mb
89	NO	991 mb
98	NO	991 mb
107	NO	991 mb
116	NO	991 mb
125	NO	991 mb
134	NO	991 mb
143	NO	991 mb
149	NO	991 mb
140	NO	991 mb
149	NO	991 mb

Sample ID: 1805008-03

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

No Finite determination. -CB 6/7/18

red lower layer. black top layer too thin to phase separate. Able to mix and emulsify top layer in bottom layer to take a representative sample aliquot.

Flash Point by AIG048A Date: 6/7/18 Analyst: CB

Continuation from previous page ↗

Sample ID: 1805008-04

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
CB 6/7/18 79 78	NO	992 mb
87	NO	992 mb
96	NO	992 mb
105	NO	992 mb
114	NO	992 mb
123	NO	992 mb
132	NO	992 mb
141	NO	992 mb
149	NO	992 mb
140	NO	992 mb
149	NO	992 mb

Sample ID: 1805008-04

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

no Finite. No Flash during range Finding. -CB 6/7/18

Sample ID: 1805008-05

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
81	NO	993 mb
90	NO	993 mb
99	NO	993 mb
108	NO	993 mb
117	NO	992 mb
126	NO	992 mb
135	NO	992 mb
144	NO	992 mb
149	NO	992 mb
140	NO	992 mb
149	NO	992 mb

Sample ID: 1805008-05

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

No Finite. No Flash during range Finding. -CB 6/7/18

Flash Point by Setaflash **Working Bench Sheet**

Work Order(s): 1805008, 1806004 Date: 6/8/18 Analyst: CB

LIMS Batch ID: B18F604

Sample ID: SRM, LIMS # 17E2502

B18F004-SRM 7

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
75	NO	996 mb
76	NO	996 mb
77	NO	996 mb
78	NO	996 mb
79	NO	996mb
80	Yes	996 mb

Sample ID: SRM, LIMS # 17E2502

B18F004-SRM8

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
77	NO	997 mb
78	NO	997 mb
79	Yes	997 mb

Sample ID: 1805008-06

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
79	NO	998 mb
88	NO	998 mb
97	NO	998 mb
106	NO	998 mb
115	NO	998 mb
123	NO	998 mb
132	NO	998 mb
141	NO	997 mb
149	NO	997 mb
140	NO	997 mb
149	NO	997 mb

Sample ID: 1805008-06

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

no Finite because no Flash during range Finding. - CB 6/8/18

Flash Point by AIG048A
Continuation from previous page

Date: 6/8/18 Analyst: CB

Sample ID: 1806004-01

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
78	NO	996 mb
87	NO	996 mb
96	NO	995 mb
105	NO	995 mb
114	NO	995 mb
123	NO	995 mb
132	NO	995 mb
141	Yes	995 mb

Sample ID: 1806004-01

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
132	NO	996 mb
133	NO	996 mb
134	NO	996 mb
135	Yes	~~996 mb~~
132	NO	997 mb
133	NO	997 mb
134	NO	997 mb
135	Yes	997 mb

996 mb CB 6/8/18

Sample ID:

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

Range Finding NA for duplicate. Finite determination duplicated. -CB 6/8/18

Sample ID: B18F004-DUP2 Source = 1806004-01

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
132	NO	997 mb
133	NO	997 mb
134	NO	997 mb
135	Yes	997 mb
132	NO	997 mb
133	NO	997 mb
134	NO	997 mb
135	Yes	997 mb

Flash Point by AIG048A

Continuation from previous page

Date: 6/8/18 Analyst: CB

Sample ID: 1806004-02

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
79	NO	996 mb
88	NO	996 mb
97	NO	996 mb
106	NO	995 mb
115	NO	995 mb
124	NO	995 mb
133	NO	995 mb
142	YES	995 mb

Sample ID: 1806004-02

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)
133	NO	995 mb
134	NO	995 mb
135	Yes	995 mb
133	NO	995 mb
134	NO	995 mb
135	Yes	995 mb

Sample ID: _____

Preliminary (Range Finding) -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

Sample ID: _____

Finite Flash Point -

Temperature (°F)	Flash (Y/N)	Pressure (specify units)

Environmental Protection Agency Region 5
Chicago Regional Laboratory
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Date: 6/13/2018

AUTOMATED ANALYZER BENCH SHEET

Batch Number: B18F011

Date: 6/13/2018

Analyst (initials): CB

Analyses Included On This Benchsheet

Water content,Karl Fisher Titration

Sample Number	Source ID	Spike Type	Spike ID	Spike Amount (uL)	Final Volume (mL)	Dilution (mL)		Comments
						Initial	Final	
1805008-01								pH = 4
1805008-02								pH = 4
1805008-03								pH = 7
1805008-04								pH = 7
1805008-05								pH = 4
1805008-06								pH = 9
1806004-01								pH = 4
1806004-02								pH = 4
1806009-01								same as B18F011-SRM1
1806009-02								same as B18F011-SRM2
1806009-03								same as B18F011-SRM3
1806009-04								same as B18F011-SRM4
B18F011-DUP1	1805008-01							

Batch Comments: updated with P&A WO 1806009.

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Date: 6/13/2018

AUTOMATED ANALYZER BENCH SHEET

Batch Number: B18F011

Date: 6/13/2018

Analyst (initials): CB

Analyses Included On This Benchsheet

Water content,Karl Fisher Titration

Sample Number	Source ID	Spike Type	Spike ID	Spike Amount (uL)	Final Volume (mL)	Dilution (mL)		Comments
						Initial	Final	
B18F011-DUP2	1805008-02							
B18F011-DUP3	1805008-03							
B18F011-DUP4	1805008-04							
B18F011-DUP5	1805008-05							
B18F011-DUP6	1805008-06							
B18F011-DUP7	1806004-01							
B18F011-DUP8	1806004-02							
B18F011-SRM1		Static	17E1709		0.05			P&A1
B18F011-SRM2		Static	17E1709		0.05			P&A2
B18F011-SRM3		Static	17E1709		0.05			P&A3
B18F011-SRM4		Static	17E1709		0.05			P&A4

Batch Comments: updated with P&A WO 1806009.

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Date: 6/13/2018

AUTOMATED ANALYZER BENCH SHEET

Batch Number: B18F011

Date: 6/13/2018

Analyst (initials): CB

Analyses Included On This Benchsheet

Water content,Karl Fisher Titration

Sample Number	Source ID	Spike Type	Spike ID	Spike Amount (uL)	Final Volume (mL)	Dilution (mL)		Comments
						Initial	Final	

PREPARATION REAGENTS/STANDARDS/PIPETTES:

17E1709:	Water Content - Hydranal Water Standard 10.0	Prepared: Jan-19-16	Expires: Dec-23-20
17E1710:	HYDRANAL - Composite 5 K	Prepared: Jul-07-16	Expires: Jun-22-19
18D1804:	HYDRANAL - Working Medium K	Prepared: Oct-04-17	Expires: Sep-24-19

Batch Comments: updated with P&A WO 1806009.

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Date: 6/12/2018

Water Content by Karl Fischer Titration

Date: 6/12/18

Analyst (initials): CB

Batch Number: B18F011

Analyses Included On This Benchsheet

Water content,Karl Fisher Titration

Sample number (LIMS ID)	Duplicate Source sample (LIMS ID)	Initial syringe weight (g)	Final syringe weight (g)	Sample weight (g)	Comments
1805008-01				0.05	pH = 4
1805008-02				0.05	pH = 4
1805008-03				0.05	pH = 7
1805008-04				0.05	pH = 7 able to emulsify top layer For representative sample.
1805008-05				0.05	pH = 4
1805008-06				0.05	pH = 9
1806004-01				0.05	pH = 4
1806004-02				0.05	pH = 4
B18F011-DUP1	1805008-01			0.05	

Batch Comments:

recorded in instrument software. -CB 6/12/18

Comments:

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Date: 6/12/2018

Water Content by Karl Fischer Titration

Date: 6/12/18

Analyst (initials): CB

Batch Number: B18F011

Analyses Included On This Benchsheet

Water content,Karl Fisher Titration

Sample number (LIMS ID)	Duplicate Source sample (LIMS ID)	Initial syringe weight (g)	Final syringe weight (g)	Sample weight (g)	Comments
B18F011-DUP2	1805008-02			0.05	
B18F011-DUP3	1805008-03			0.05	
B18F011-DUP4	1805008-04			0.05	
B18F011-DUP5	1805008-05			0.05	
B18F011-DUP6	1805008-06			0.05	
B18F011-DUP7	1806004-01			0.05	
B18F011-DUP8	1806004-02			0.05	
B18F011-SRM1	-			0.05	P&A1
B18F011-SRM2	-			0.05	P&A2

Batch Comments:

CB 6/12/18
recorded in software.

Comments:

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Phone:(312)353-8370 Fax:(312)886-2591

Date: 6/12/2018

Water Content by Karl Fischer Titration

Date: 6/12/18

Analyst (initials): CB

Batch Number: B18F011

Analyses Included On This Benchsheet

Water content,Karl Fisher Titration

Sample number (LIMS ID)	Duplicate Source sample (LIMS ID)	Initial syringe weight (g)	Final syringe weight (g)	Sample weight (g)	Comments
B18F011-SRM3	-			0.05	P&A3
B18F011-SRM4	-			0.05	P&A4

PREPARATION REAGENTS/STANDARDS/PIPETTES:

17E1709: Water Content - Hydranal Water Standard 10.0

17E1710: HYDRANAL - Composite 5 K

18D1804: HYDRANAL - Working Medium K

CB 6/12/18
recorded in instrument software.

Batch Comments:

Comments:

Balance #21 logbook

	Standard Weight	Actual Weight
12/28/17	0.1g	0.1000g
CB	0.5g	0.5001g
Weight Set	1g	1.0000g
Troemner	5g	4.9999g
S/N 40000017548	10g	10.0001g
	50g	49.9999g
	100g	100.0000g

CB 1/9/18 • Balance OK after building water damage.

CB 5/17/18	Standard Weight	Reading
Weight Set	0.1g	0.0999g
Troemner	0.5g	0.5000g
S/N 40000020557	1g	1.0000g
	5g	5.0000g
	10g	9.9999g
	50g	50.0001g
	100g	100.0001g

CB 6/6/18	0.1g	0.1000g
Weight Set	0.5g	0.5000g
Troemner	1g	1.0001g
S/N 40000020557	5g	5.0000g
	10g	10.0000g
	50g	49.9999g
	100g	99.9998g

20

	Standard Weight	Actual Weight
CB 6/7/18	0.1g	0.1000g
Weight Set	0.5g	0.4999g
Troemner	1g	0.9999g
S/N 40000 20557	5g	5.0000g
	10g	10.0000g
	50g	50.0000g
	100g	100.0000g

CB 6/8/18

~~66~~ CB 6/8/18

Weight Set	0.1g	0.1000g
Troemner	0.5g	0.5000g
S/N 40000 20557	1g	0.9999g
	5g	5.0001g
	10g	10.0001g
	50g	50.0000g
	100g	99.9999g

CB 6/12/18	0.1g	0.1000g
Weight set	0.5g	0.4999g
Troemner	1g	1.0001g
S/N 40000 20557	5g	5.0000g
	10g	10.0000g
	50g	50.0001g
	100g	99.9999g

KFT instrument logbook

CB 6/14/17
- Analyzed B17F023.
- System ran well, only issues with difficult sample matrices were encountered. Titer strength, conditioning response, and Hydranal 10.0 (LIMS 17E1709) all performed well and/or met QC limits.

CB 6/15/17
- Analyzed B17F024
- Titer strength determination took multiple attempts. Cleaned apparatus and repeated until passing.

CB 7/26/17
- Analyzed B17G047
- System ran well. Conditioning was good.

CB 12/28/17
- Pump to Fill titration vessel was not working. Opened 803 Ti Stand housing and tightened down switch. Filling pump began working.
- Analyzed B17L033 CB 12/28/17
- Titre strength determination required ~~to~~ two attempts. Afterwards conditioning was successful and run was OK.

CB 1/9/18
- Instrument and computer OK after building water damage. Balance #21 OK too.

CB 6/12/18
- Analyzed B18F011. System Ran Well.

(6)

Setaflash 3 instrument logbook

5/17/18 CB
- Re-installed Thermometer (S/N85Cϕ2ϕ exp 9/15/18) after being out ~~for~~ calibration.
- Syringe check. Syringe ID = N-1244. Balance #21. 1.9414 g. OK at 2.0 ± 0.1 mL.
- Barometer ID = LIMS 17C1307.

6/6/18 CB
- Syringe ID = N-1244
- Syringe Check on Balance #21, 1.9518 g. OK at 2.0 ± 0.1 mL.
- Barometer ID = LIMS 17C1307

6/7/18 CB
- Syringe ID = N-1244
- Balance #21
- 1.9272 g → OK at 2.0 ± 0.1 mL.
- Barometer ID = LIMS 17C1307

6/8/18 CB
- Syringe ID = N-1244
- Balance #21
- 1.9420 g → OK at 2.0 ± 0.1 mL.
- Barometer ID = LIMS 17C1307

ANALYSIS SEQUENCE

Printed: 6/13/2018 11:40:30AM

18F1302

Instrument: KFT **Analysis Date:** Jun-12-18
Calibration ID: UNASSIGNED **Matrix:** Water

Order	Container	Lab Number	Sample name or QC sample designation	Analysis	STD ID	Comments
1		B18F011-SRM1	Reference	QC		
2	A	1806009-01	P&A1	Water content,Karl Fisher Titration		
3		B18F011-SRM2	Reference	QC		
4	A	1806009-02	P&A2	Water content,Karl Fisher Titration		
5		B18F011-SRM3	Reference	QC		
6	A	1806009-03	P&A3	Water content,Karl Fisher Titration		
7		B18F011-SRM4	Reference	QC		
8	A	1806009-04	P&A4	Water content,Karl Fisher Titration		
9	C	1805008-02	HCC02	Water content,Karl Fisher Titration		pH = 4
10		B18F011-DUP2	Duplicate	QC		
11	B	1806004-01	01 Tetramer	Water content,Karl Fisher Titration		pH = 4
12		B18F011-DUP7	Duplicate	QC		
13	B	1806004-02	02 Tetramer	Water content,Karl Fisher Titration		pH = 4
14		B18F011-DUP8	Duplicate	QC		
15	B	1805008-01	HCC01	Water content,Karl Fisher Titration		pH = 4
16		B18F011-DUP1	Duplicate	QC		
17	B	1805008-03	HCC03	Water content,Karl Fisher Titration		pH = 7
18		B18F011-DUP3	Duplicate	QC		
19	C	1805008-04	HCC04	Water content,Karl Fisher Titration		pH = 7
20		B18F011-DUP4	Duplicate	QC		
21	C	1805008-05	HCC05	Water content,Karl Fisher Titration		pH = 4
22		B18F011-DUP5	Duplicate	QC		

Order	Container	Lab Number	Sample name or QC sample designation	Analysis	STD ID	Comments
23	B	1805008-06	HCC06	Water content,Karl Fisher Titration		pH = 9
24		B18F011-DUP6	Duplicate	QC		

PREPARATION REAGENTS/STANDARDS/PIPETTES:

17E1709: **Water Content - Hydranal Water Standard 10.0** **Prepared: Jan-19-16** **Expires: Dec-23-20**

REVIEWED

By Colin Breslin at 11:41 am, Jun 13, 2018

Analyst Initials and Date

Determination overview

	Determination start	Sample ID	Method name	Titer (mg/mL)	KF Titer Mean value	Titer s(rel)	% Water	mg Water	Sample size (g)	mL to EP	Remarks
1	2018-06-12 12:20:35 UTC-5	B18F011-DUP6	KF SAMPLE				96.29	962.94	-0.0512	10.25	
2	2018-06-12 12:17:51 UTC-5	1805008-06	KF SAMPLE				96.06	960.57	-0.0606	12.10	
3	2018-06-12 12:13:33 UTC-5	B18F011-DUP5	KF SAMPLE				0.05	0.50	-4.2541	0.44	
4	2018-06-12 12:10:12 UTC-5	1805008-05	KF SAMPLE				0.05	0.50	-4.0966	0.42	
~~5~~	~~2018-06-12 12:06:35 UTC-5~~	~~1805008-05~~	~~KF SAMPLE~~				~~0.26~~	~~2.57~~	~~-0.0412~~	~~0.02~~	
6	2018-06-12 12:01:26 UTC-5	B18F011-DUP4	KF SAMPLE				55.34	553.43	-0.0591	6.80	1
7	2018-06-12 11:56:50 UTC-5	1805008-04	KF SAMPLE				55.58	555.77	-0.0615	7.11	
8	2018-06-12 11:52:39 UTC-5	B18F011-DUP3	KF SAMPLE				96.84	968.37	-0.0423	8.52	
9	2018-06-12 11:49:34 UTC-5	1805008-03	KF SAMPLE				96.49	964.94	-0.0425	8.53	
~~10~~	~~2018-06-12 11:43:39 UTC-5~~	~~1805008-03~~	~~KF SAMPLE~~				~~invalid~~	~~invalid~~	~~-1.0466~~	~~invalid~~	
11	2018-06-12 11:40:34 UTC-5	B18F011-DUP1	KF SAMPLE				0.01	0.12	-4.2388	0.10	2
12	2018-06-12 11:37:21 UTC-5	1805008-01	KF SAMPLE				0.01	0.10	-3.9470	0.08	
~~13~~	~~2018-06-12 11:34:18 UTC-5~~	~~1805008-01~~	~~KF SAMPLE~~				~~0.02~~	~~0.18~~	~~-0.8232~~	~~0.03~~	
14	2018-06-12 11:26:15 UTC-5	B18F011-DUP8	KF SAMPLE				0.01	0.09	-3.9783	0.07	3
15	2018-06-12 11:23:07 UTC-5	1806004-02	KF SAMPLE				0.01	0.08	-3.8427	0.06	
16	2018-06-12 11:18:23 UTC-5	B18F011-DUP7	KF SAMPLE				0.01	0.10	-3.8794	0.08	
17	2018-06-12 11:14:52 UTC-5	1806004-01	KF SAMPLE				0.01	0.09	-3.8999	0.07	
~~18~~	~~2018-06-12 11:10:27 UTC-5~~	~~1806004-01~~	~~KF SAMPLE~~				~~0.01~~	~~0.12~~	~~-0.7999~~	~~0.02~~	
19	2018-06-12 11:02:50 UTC-5	B18F011-DUP2	KF SAMPLE				1.16	11.58	-0.7991	1.92	4
20	2018-06-12 10:57:30 UTC-5	1805008-02	KF SAMPLE				1.05	10.52	-0.7945	1.74	
21	2018-06-12 10:51:16 UTC-5	Hydranal 10.0 (B18F011-SRM4)	KF SAMPLE				1.04	10.44	-1.0028	2.18	
22	2018-06-12 10:47:37 UTC-5	Hydranal 10.0 (B18F011-SRM3)	KF SAMPLE				1.03	10.30	-1.0454	2.24	
23	2018-06-12 10:44:22 UTC-5	Hydranal 10.0 (B18F011-SRM2)	KF SAMPLE				1.04	10.39	-1.0347	2.23	
24	2018-06-12 10:33:39 UTC-5	Hydranal 10.0 (B18F011-SRM1)	KF SAMPLE				1.04	10.41	-1.0436	2.26	
25	2018-06-12 10:27:18 UTC-5	Water	KF TITER	4.85	4.81	0.72			-0.0521	10.74	
26	2018-06-12 10:23:37 UTC-5	Water	KF TITER	4.79	4.79	0.00			-0.0529	11.04	
27	2018-06-12 10:20:16 UTC-5	Water	KF TITER	4.79	4.79	invalid			-0.0361	7.54	

REVIEWED
By Colin Breslin at 12:19 pm, Jun 13, 2018

5

Summary of Comments on Titel

Page: 1

Number: 1 Author: cbreslin Subject: Inserted Text Date: 6/13/2018 11:34:31 AM
repeated with larger sample size.

Number: 2 Author: cbreslin Subject: Inserted Text Date: 6/13/2018 11:33:40 AM
over range. repeated with smaller sample size.

Number: 3 Author: cbreslin Subject: Inserted Text Date: 6/13/2018 11:32:55 AM
repeated with larger sample size.

Number: 4 Author: cbreslin Subject: Inserted Text Date: 6/13/2018 11:32:16 AM
repeated with larger sample size.

Number: 5 Author: cbreslin Subject: Reviewed Date: 6/13/2018 12:19:51 PM

Flash Point Calculations

Instructions for Use:

1) Enter the unique sample ID under "LIMS ID".
2) Enter the observed flash point temp (°F) under "Flash Point Temp Recorded (°F)".
3) Enter the observed barometric pressure under the appropriate column to match the units recorded.
4) The spreadsheet automatically calculates the barometrically corrected flash point based on the units of pressure used.
4) The final calculated result is given under "Calculated Flash Point (°F).
5) Follow CRL SOP AIG048A for reporting the final results.

Date of Analysis: 6/6/2018 Analyst: CB

Work Order(s): 1805008 LIMS Batch ID: B18F004

Count	LIMS ID	Flash Point Temp Recorded (°F)	Barometric Pressure Recorded (mm Hg)	Barometric Pressure Recorded (mb)	Barometric Pressure Recorded (in Hg)	Calculated Flash Point (°F)
1	B18F004-SRM1	79		990.0		80.05
2	B18F004-SRM2	80		990.0		81.05
				B18F004-SRM3 (p-Xylene Phillips 66), average =		**80.55**
3	1805008-02	145		991.0		146.00
4	1805008-02	146		990.0		147.05
				1805008-02, average =		**146.52**
5	B18F004-DUP1	147		988.0		148.14
6	B18F004-DUP1	147		988.0		148.14
				B18F004-DUP1, average =		**148.14**

Flash Point Calculations

Instructions for Use:

1) Enter the unique sample ID under "LIMS ID".
2) Enter the observed flash point temp (°F) under "Flash Point Temp Recorded (°F)".
3) Enter the observed barometric pressure under the appropriate column to match the units recorded.
4) The spreadsheet automatically calculates the barometrically corrected flash point based on the units of pressure used.
4) The final calculated result is given under "Calculated Flash Point (°F).
5) Follow CRL SOP AIG048A for reporting the final results.

Date of Analysis: 6/7/2018 Analyst: CB

Work Order(s): 1805008 LIMS Batch ID: B18F004

Count	LIMS ID	Flash Point Temp Recorded (°F)	Barometric Pressure Recorded (mm Hg)	Barometric Pressure Recorded (mb)	Barometric Pressure Recorded (in Hg)	Calculated Flash Point (°F)
1	B18F004-SRM4	80		990.0		81.05
2	B18F004-SRM5	80		990.0		81.05
	B18F004-SRM6 (p-Xylene Phillips 66), average =					**81.05**
3						45.60
4						45.60

Flash Point Calculations

Instructions for Use:

1) Enter the unique sample ID under "LIMS ID".
2) Enter the observed flash point temp (°F) under "Flash Point Temp Recorded (°F)".
3) Enter the observed barometric pressure under the appropriate column to match the units recorded.
4) The spreadsheet automatically calculates the barometrically corrected flash point based on the units of pressure used.
4) The final calculated result is given under "Calculated Flash Point (°F).
5) Follow CRL SOP AIG048A for reporting the final results.

Date of Analysis: 6/8/2018 Analyst: CB

Work Order(s): 1805008, 1806004 LIMS Batch ID: B18F004

Count	LIMS ID	Flash Point Temp Recorded (°F)	Barometric Pressure Recorded (mm Hg)	Barometric Pressure Recorded (mb)	Barometric Pressure Recorded (in Hg)	Calculated Flash Point (°F)
1	B18F004-SRM7	80		996.0		80.78
2	B18F004-SRM8	79		997.0		79.73
				B18F004-SRM9 (p-Xylene Phillips 66), average =		**80.25**
3	1806004-01	135		996.0		135.78
4	1806004-01	135		997.0		135.73
					1806004-01, average =	**135.75**
5	B18F004-DUP2	135		997.0		135.73
6	B18F004-DUP2	135		997.0		135.73
					B18F004-DUP2, average =	**135.73**
7	1806004-02	135		995.0		135.82
8	1806004-02	135		995.0		135.82
					1806004-02, average =	**135.82**

Precision and Accuracy/IDOC Study

Analyst: Colin Breslin
Analysis Date: 6/12/2018
SOP No. and Version: AIG015A V4
SOP Name: Water Content by Karl Fischer Titration
Current Limits: 97 - 107 %

To use template for calculations, save the sheet as another name and enter the information needed. Change units and significant digits based on SOP.

Sample LIMS ID	Spiked Conc. (%)	Measured Conc. (%)	% Recovery
B18F011-SRM1	1.00	1.04	104.0
B18F011-SRM2	1.00	1.04	104.0
B18F011-SRM3	1.00	1.03	103.0
B18F011-SRM4	1.00	1.04	104.0

CALCULATIONS								
n	$\bar{x}$	s	2s	3s	$\bar{x}$ - 2s	$\bar{x}$ + 2s	$\bar{x}$ - 3s	$\bar{x}$ + 3s
4	1.0375	0.0050	0.0100	0.0150	1.0275	1.0475	1.0225	1.0525
4	103.8	0.500	1.0	1.5	102.8	104.8	102.3	105.3

Where:

n = Number of replicate analyses
s = Sample (n-1) standard deviation of the replicate analyses
2s = Warning limits for precesion control chart
3s = Action limits for precision control chart
$\bar{x}$ = Mean of the replicate analyses
$\bar{x}$ ± 2s = Warning limits for accuracry control chart
$\bar{x}$ ± 3s = Action limits for accuracy control chart

Version 1
Modified 6/1/2011
Calculations Reviewed by Greg Mitsakopoulos 6/3/11